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NATIONAL BUREAU OF STANDARDS

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# Technical News Bulletin

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TECHNOLOGY & SCIENCE



U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

# Technical News Bulletin

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U.S. DEPARTMENT OF COMMERCE

John T. Connor, Secretary

NATIONAL BUREAU OF STANDARDS

A. V. Astin, Director

## CONTENTS

- 149 Ceramic semiconductors found superconducting
- 151 Office of engineering standards liaison and analysis established at NBS
- 152 Cryogenic cooler for semiconductor devices
- 154 Research on archival microfilm
- 156 Effect of high pressure on refractive index
- 157 Standards and calibration
  - U.S. standard frequency and time broadcasts
- 158 State invention shows aid industry
- 161 Construction of new WWV buildings begun
- 162 Accurate peak pulse voltage instruments for low and medium ranges
- 163 NMR calibration of permanent magnet standards
- 164 Precise technique measures absolute reflectance
- 165 Symposium on ceramic materials
- 166 NSRDS news
- 168 Publications of the National Bureau of Standards



## COVER

*Blemishes form on microfilm as shown by this concentric ring structure photographed at NBS with a high power microscope. This ring is typical of type I aging blemishes. (See story on p. 154.)*

Prepared by the NBS Office of Technical Information and Publications

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The National Bureau of Standards serves as a focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. For this purpose, the Bureau is organized into three institutes—

- The Institute for Basic Standards
- The Institute for Materials Research
- The Institute for Applied Technology

The TECHNICAL NEWS BULLETIN is published to keep science and industry informed regarding the technical programs, accomplishments, and activities of all three institutes.

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*E. Pfeiffer moves the external field magnet up around the magnetometer after the apparatus has been cooled below the transition temperature of the ceramic specimen in the magnetometer. This magnet produces fields up to 1500 Oe at the ceramic superconductor.*



## CERAMIC SEMICONDUCTORS FOUND SUPERCONDUCTING

■ New knowledge of the superconducting state of matter has been obtained in research on semiconducting materials at the National Bureau of Standards. In a study supported partly by the Advanced Research Projects Agency and the National Aeronautics and Space Administration, transitions to the superconducting state were produced in ceramic samples of strontium titanate and in mixed-composition ceramic samples of barium-strontium and calcium-strontium titanates. This is believed to be the first time that ceramic semiconductors have been made superconduct-

ing. The investigation was conducted by H. P. R. Frederikse, J. F. Schooley, W. R. Thurber, E. Pfeiffer, and W. R. Hosler of the Bureau staff.<sup>1</sup>

The NBS findings are especially significant because they indicate that by varying the composition of semiconducting materials it is possible to control the essential parameters of the superconducting state in these materials. Such control could be helpful in the development of superconducting materials having properties tailored to specific applications as in high-field magnets, superspeed switches in compact computers, and

magnetic shields in spacecraft.

In 1964, J. F. Schooley and W. R. Hosler of NBS showed<sup>2</sup> that oxide-type semiconducting single crystals could be made superconducting. In particular, reduced strontium titanate was found to be a superconductor with a transition temperature in the range 0.03 to 0.25 °K. The variation in transition temperature depended upon the amount of reduction in the strontium titanate (ranging from slightly reduced to fully reduced) and confirmed theoretical predictions by M. L. Cohen<sup>3</sup> about the electronic state in the semiconductor necessary

*continued*

## SEMICONDUCTORS *continued*

for transition. In his theory Dr. Cohen\* also indicated that lattice configurations have an influence on the transition temperature of a superconductor. Changes in the lattice system of an oxide-type semiconductor, such as strontium titanate, are relatively easy to make by the partial substitution of calcium or barium for the strontium. Thus, doped strontium titanate was considered an ideal material for additional study of the superconducting state.

Because of the difficulty of obtaining single crystals of strontium titanate with large amounts of substituted calcium or barium in their structure, ceramic specimens prepared by H. Johnson of the NBS crystal chemistry laboratory were used in the present study. These specimens were fabricated by compressing powdered material into pellets and heating them to a temperature of 1650 °C. This treatment was required to insure that the grain sizes in the ceramic samples would be 20 microns or larger. Otherwise the magnetic fields that were used to test for superconductivity would penetrate through the grains and destroy the superconductivity of the sample.

Superconducting transition temperatures are found by measuring the temperature where the electrical resistance becomes zero or by determining the magnetic moment or magnetic susceptibility of the superconductor. In this investigation the magnetic moment was determined with a vibrating-coil magnetometer developed at NBS.<sup>4</sup> With this instrument the magnetic moment of the superconductor was measured at various temperatures as a function of the applied magnetic field. A plot of the critical field (the magnetic field where superconductivity breaks down) against temperature gave the transition temperature. Magnetic susceptibility measurements were used in a similar way.

When the prepared ceramic speci-

mens of strontium titanate were tested for superconductivity, they were found to have transition temperatures agreeing closely with those of single crystal specimens that had the same electron concentration. Essentially, this means that the samples were at the same level of reduction.

The ceramic specimens of doped strontium titanate were prepared with a 0–20 percent substitution of barium and a 0–100 percent substitution of calcium. When these samples were tested for superconductivity, the barium-doped strontium titanate was superconducting up to approximately 12 percent added barium. The transition temperature increased, as the barium content increased, to a maximum of 0.55 °K at 5 percent added barium, and then decreased to less than 0.1 °K at 12 percent added barium. Calcium-doped strontium titanate, however, exhibited superconductivity up to approximately 35 or 45 percent added calcium. As the calcium content increased, the transition temperature increased to a maximum temperature of 0.55 °K at 30 percent added calcium. These maximum transition temperatures were nearly twice that of the undoped strontium titanate.

Future work at NBS will investigate superconducting transitions in semiconducting materials that have a crystalline structure similar to that of strontium titanate. Such investigations may provide additional knowledge of the superconducting state.

<sup>1</sup> For further technical details, see Superconductivity in ceramic, mixed titanates, by H. P. R. Frederikse, J. F. Schooley, W. R. Thurber, E. Pfeiffer, and W. R. Hosler, *Phys. Rev. Letters* 16, 579 (1966).

<sup>2</sup> Superconductivity observed in the semiconductor strontium titanate, NBS Tech. News Bull. 48, 126–127 (1964). Also, Superconductivity in semiconducting  $\text{SrTiO}_3$ , by J. F. Schooley, W. R. Hosler, and M. L. Cohen, *Phys. Rev. Letters* 12, 474 (1964).

<sup>3</sup> Superconductivity in many-valley semiconductors and in semimetals, by M. L. Cohen, *Phys. Rev.* 134, A511 (1964).

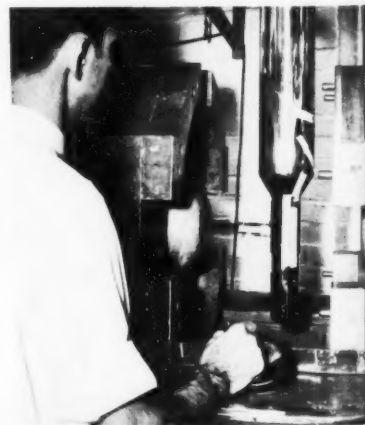
<sup>4</sup> Vibrating-coil magnetometer for use at very low temperatures, by R. S. Kaeser, E. Ambler, and J. F. Schooley, *Rev. Sci. Instr.* 37, 173 (1966).

\*Present address: Department of Physics, University of California, Berkeley, Calif.



E. Pfeiffer inserts a specimen mount in the vibrating-coil magnetometer. This apparatus measures the magnetic critical field of the superconducting ceramic, doped-strontium titanate. The doped-strontium titanate specimen is in a small container just above Mr. Pfeiffer's left hand.

With the specimen in the Dewar, the cryostat is moved between the pole faces of a high-field magnet. The magnet, which operates at fields up to 20 kOe, cools the ceramic to 0.06 °K by adiabatic demagnetization.



## OFFICE OF ENGINEERING STANDARDS LIAISON AND ANALYSIS ESTABLISHED AT NBS

*Dr. George S. Gordon Appointed Chief*



*Dr. George S. Gordon*

■ The National Bureau of Standards has established an Office of Engineering Standards Liaison and Analysis to serve as a focal point for NBS assistance to engineering standards-making bodies throughout the Nation. The new Office will aid in making the Bureau's technical resources available to strengthen private standards organizations, manufacturers, and government agencies concerned with the development of performance standards and test methods for industrial products. It will be headed by Dr. George S. Gordon, who previously was Chief of the Textile and Apparel Technology Center of the NBS Institute for Applied Technology.

For more than 60 years the National Bureau of Standards has played a key supporting role in the development of engineering standards—specifications, standards of practice, and methods of test—for technological devices, products, and services. Most of this work has been done in cooperation with technical societies and standardization groups such as the American Standards Association and the American Society for Testing and Materials. Normally the Bureau assists by providing expert advice and technical assistance on both national and international engineering standards committees.

In recent years there has been a growing awareness of the importance of engineering standards, both domestic and international, to the economic development of the country. This feeling has been strengthened within the past year by the publication of the recommendations of the Department of Commerce Panel on Engineering and Commodity Standards, known as the LaQue Committee.<sup>1</sup> Among other things, the Panel recommended greater participation by Federal agencies in certain aspects of the development of engineering standards by nongovernmental organizations, recognition of the importance that U.S. industry's participation in international standardization can have on the national economy, and communication of the importance of such participation to industry.

The Office of Engineering Standards Liaison and Analysis will aid in the achievement of these objectives through a program of active liaison with other standardizing organizations and by general review and analysis of the engi-

neering standardization activities of the Bureau staff. It will endeavor to find new ways to increase the effectiveness of NBS technical assistance in the development of engineering standards and to match NBS technical resources to the needs of outside organizations. It will also make a continuous effort to extend and improve communication between the Bureau and other engineering standards bodies so that NBS may know and respond to their needs and so that NBS capabilities in various technical fields may be better known to them.

The new Office will be a part of the NBS Director's Office, but it will work closely with the Manager of Engineering Standards, Malcolm W. Jensen, in the NBS Institute for Applied Technology. The Manager of Engineering Standards is responsible for NBS cooperation with producers, distributors, consumers, and government agencies in the development of product standards—voluntary trade standards that define quality levels of commercial products and hold variety in such products to a minimum. He is also responsible for the development and evaluation of those safety standards (such as automotive seat belt standards) that are required by statute, and the provision of information services on engineering standards.

Dr. Gordon has been a member of the Bureau staff since June 1964. For 5 years previously he was Vice President and Director of Chemistry Research at IIT Research Institute. From 1955 to 1958 he was Director of Research for the United Potash Co., and Associate Director of Research for the U.S. Borax Research Corp. in Los Angeles. In 1951 he helped to found the Titanium Zirconium Co. and remained as Vice President until 1955.

Born in Pittsfield, Mass., Dr. Gordon completed an A.B. in chemistry at Princeton University and a Ph. D. in chemistry at Northwestern University. He is a member of Phi Beta Kappa, Sigma Xi, and the American Chemical Society.

<sup>1</sup> See Standardization study released, NBS Tech. News Bull, 49, 82 (May 1965). The full report is available from the Clearinghouse for Federal Scientific and Technical Information, U.S. Department of Commerce, Springfield, Va. 22151. Section A, price \$2, No. PB 166811; Section B, price \$6, No. PB 166812. Section A contains major findings and recommendations. Section B contains reports of the various task forces.

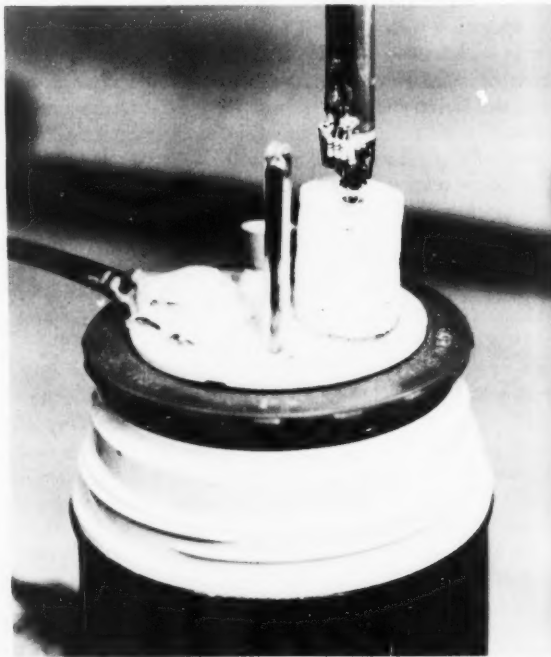


# CRYOGENIC COOLER FOR SEMICONDUCTOR DEVICES

■ Precisely controlled cryogenic ambients are obtained in the NBS Institute for Applied Technology by use of a laboratory cooler devised there by physicist Herbert K. Kessler.<sup>1</sup> The cooler removes heat developed within semiconductor devices to permit operation at low temperatures. It is particularly useful in investigations involving light emitters, lasers, and photodetectors because it enables them to be operated at cryogenic temperatures without being enclosed; otherwise optical windows would be required. Because the cooler operates by directing a stream of cold gas on the cooled device, it can supply controlled cooling to a variety of laboratory setups.

Many equipments in and outside the laboratory (internal combustion motors, nuclear generators, and electronic devices, for instance) generate so much heat that systems to remove it must be built in to prevent damage and to permit efficient operation. Most often this is done by directing a flow of ambient air around the hot parts or by circulating cooling liquids around them.

*Left: the Dewar flask is removed by Herbert Kessler from a cryogenic cooler. This reveals the gas inlet tube, heat transfer coils, outlet tube, level indicator float, and heater. Small devices to be cooled are operated within the frost shield surrounding the nozzle. Right: a laser supported from its transmission line is cooled during operation by a stream of cold gas (not visible) flowing around it. The gas, generated in the cooler, is supplied at a rate which can be controlled electrically.*



Semiconductor devices (light emitters, lasers, and photodetectors) which must be operated at cryogenic temperatures are usually mounted on thermally conducting "cold fingers" having one end in liquefied gas, or sometimes are completely immersed in the liquid. However, the cold-finger method is unsuitable for devices producing large amounts of power because of the large thermal gradients that are created. The immersion method is also of limited usefulness because of thermal shock, which can shatter the device, and because of bubbling, which destroys thermal contact between device and coolant and also interferes with the optical path. A cooling system which avoids these shortcomings and which does not require a Dewar enclosure with optical windows will be useful in experimentation with semiconductor and other devices.

### Design of the Cooler

The cooler designed at NBS uses the pressure of the cryogenic gas to force the gas to flow through the cooler and out to the device being cooled. Its construction is simplified by mounting parts on the insulating stopper of the Dewar flask containing the liquefied gas; coolant flow begins when the stopper assembly is pressed in place and current supplied to the heater. The parts supported in this way are a float-type level indicator, the gas outlet and heat exchanger tubing, and a small electrical heater to increase the gas pressures.

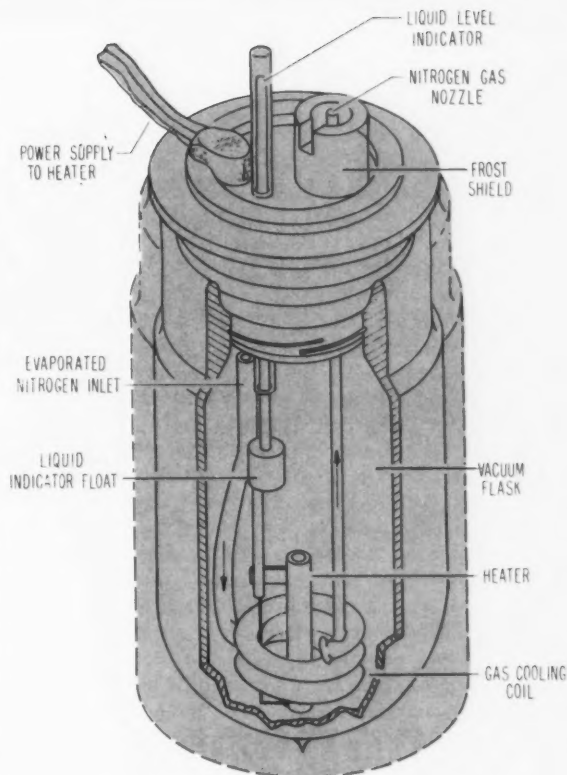
Gas pressure from evaporation at the liquid's surface forces gas at the top of the flask into copper tubing descending to a heat exchange coil beneath the surface of the liquid. There it is reduced nearly to the temperature of the liquefied gas and then flows up and out of the cooler through a nickel exit tube. It escapes through a small nozzle directed at the device being cooled.

### Use of the Cooler

A small plastic-foam frost shield surrounds the nozzle and the device under test, thus confining the cooling effect. The heater power is adjusted between 5 and 20 watts to produce gas flow at the rate desired—sufficient for the dissipation, size, and configuration of the device being cooled. More power to the heater produces more evaporation and increased flow rate.

In one application the cold gas stream impinges directly on a solid state laser supported at the end of its transmission line just inside the frost shield. The shield prevents accumulation of frost at the nozzle, directs the flow around the gallium arsenide laser, and has a cutout in a side through which the laser's beam emerges. The nozzle used can be shaped in each application for best cooling of the device.

Larger semiconductor devices are mounted in a second Dewar to which gas is piped from the cooler. The device is supported on a thermally insulating stopper closing the open end of the Dewar, which is fitted with appropriate



*In the cryogenic cooler nitrogen gas enters tubing, cooled to the liquid's temperature, and escapes as gas through a nozzle inside the frost shield. The rate of gas flow is controlled by the heater current; its temperature is close to that of the cryogenic liquid unless heated in the exhaust tubing.*

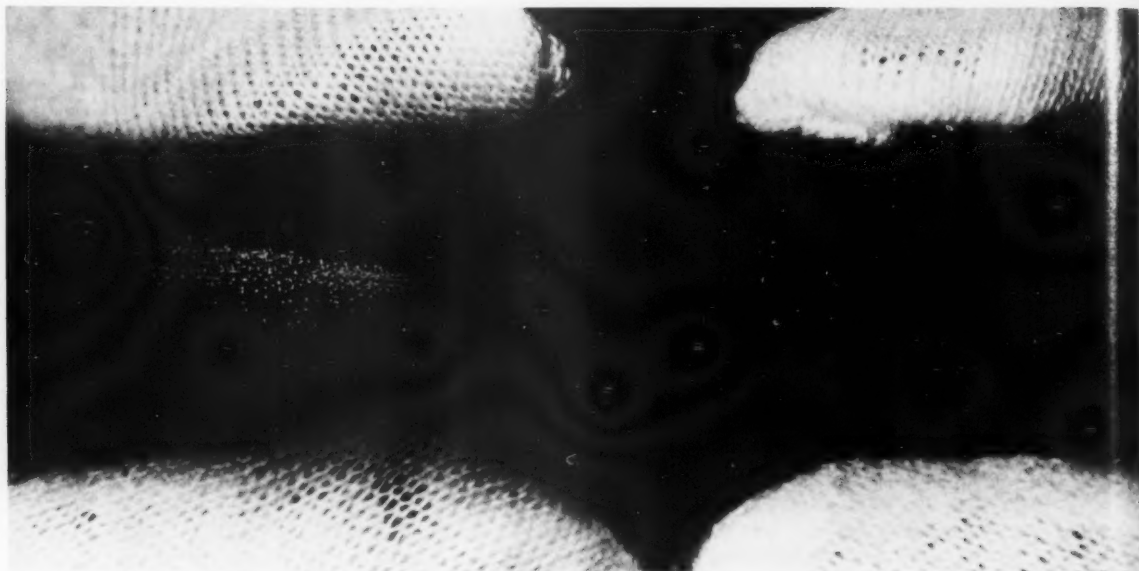
windows at the other end. The inlet and outlet tubes pass through the stopper and are located so as to provide effective cooling.

### Design Variations

By using additional heaters, the temperature of the gas issuing from the nozzle can be controlled over a range from that of liquid nitrogen to above room temperature. These heater coils are installed in the outlet pipe of the heat exchanger or in tubing outside the cooler; the power applied to them is regulated to obtain temperatures that are not directly available from cryogenic liquids.

The cooler is charged by removing the stopper and pouring in the liquid gas, a pint of nitrogen being sufficient for 35 to 75 minutes of typical operation. For operation over long periods, the cooler can be refilled while in use through a spout (normally capped) passed through the stopper.

<sup>1</sup> Cooler for semiconductor light emitters, lasers, and photodetectors, by H. K. Kessler, *Rev. Sci. Inst.* **37**, 517-518 (April 1966).



*Some aging blemishes may be seen on a piece of 16 mm film held between the gloved thumb and forefingers of an inspector.*

# Research on Archival Microfilm

guarding against blemishes

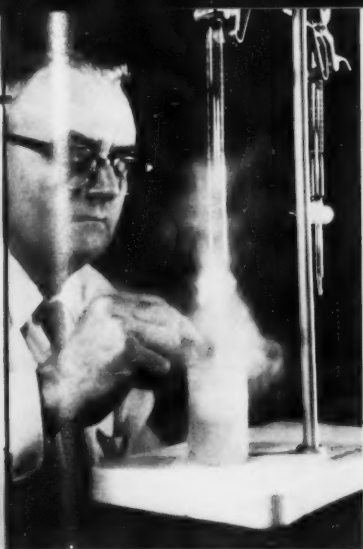
■ Scientists at the NBS Institute for Basic Standards are investigating aging blemishes that sometimes form on microfilm years after the films are placed in storage. This research<sup>1</sup> is being conducted by C. S. McCamy, C. I. Pope, and D. R. Lehmbeck, of the Institute's Photographic Research Section, and is partly sponsored by the photographic industry.<sup>2</sup> Although the details of the mechanism of blemish formation are not yet fully understood, investigations to date have revealed enough data about causative factors to permit NBS to make tentative recommendations for processing, storage, and inspection procedures.

Microfilm is widely used to preserve records of permanent value because the information can be copied in a much reduced size and thus stored in a small space. In addition, the photographic copy is generally more permanent than the original paper record.

The discovery of aging blemishes on some microfilm has generated extensive research into the causes of blemishes both at NBS and throughout the photographic industry. The objective of these studies is to obtain a clear understanding of the physics and chemistry of the reaction so that proper measures may be taken to prevent blemish formation.

The investigations have followed four general lines: detailed statistical studies of the findings of about 100 Federal microfilm inspectors, extensive field investigations of processing and storage conditions, analytical studies of films and processing solutions, and accelerated aging tests





*Left: Chester I. Pope, NBS photographic chemist, titrates hydrogen peroxide into a solution of silver salts and chloride ions. Such experiments have helped in formulating the current theory of the chemistry of blemish formation. Right: a sample of microfilm, sandwiched between glass plates, is placed in a constant-humidity test chamber to accelerate reactions occurring in natural storage. Reactants are liberated from specially treated strips of paper in the two small beakers. Under the conditions of the test, results may be obtained in less than a day.*

under a large variety of conditions in the presence of various suspected causative agents.

Research at NBS, at the Armed Forces Institute of Pathology, and at the Kodak Research Laboratories has led to the conclusion that biological mechanisms are not directly contributory. All findings thus far indicate the spot formation is a chemical process, and as usual in such cases, the probability of reaction depends on many factors. These include the exposure and processing conditions as well as various aspects of film storage.

### Exposure and Processing

In exposing and processing microfilm, NBS recommends adherence to the current American Standards pertinent to microfilming, with minor exceptions. Processors should use the least density that will produce satisfactory results, rather than strive for a high density for appearance sake, as blemish formation is more likely on high density areas than on low density areas. Some evidence indicates that it is desirable to add 0.2 gram of potassium iodide to each liter of fixing solution not otherwise containing an equivalent amount of iodide ions.<sup>3</sup>

Processors should be careful to insure that the film is thoroughly washed to remove chemicals remaining after processing, as failure to do so invites general fading reactions and spot formation. The efficient removal of water droplets before the drying operation, uniform drying, and general cleanliness are, of course, essential. Also, scratching the film should be avoided, not only because scratches are undesirable in themselves, but because blemishes form along scratches.

### Storage Conditions

Archival films should not be exposed to sulfur dioxide, hydrogen sulfide, peroxides, volatile bases such as ammonia, volatile acids such as acetic acid, engine exhaust gases, or gaseous industrial wastes. The fumes from turpentine, linseed oil, or drying paints containing such mate-

rials, should also be avoided in microfilm storage areas. To eliminate the peroxide generated in paper cartons and to protect films from gases and vapors originating elsewhere, microfilms should be stored in sealed containers impermeable to peroxides and other such vapors.

For inactive storage, microfilm should be sealed in equilibrium with air at a relative humidity of 15 to 20 percent at 50 to 60 °F. For active files, film should be sealed in equilibrium with air at a relative humidity of 30 to 35 percent at 50 to 60 °F. The storage temperature should not be permitted to exceed 70 °F and it should be maintained between 50 and 60 °F where practicable.

The probability of information loss can be very greatly reduced by making more than one microfilm copy and storing these copies in separate places. When additional security is desired, positive films should be retained, as no blemishes have been observed on the information areas of positive prints having dark characters on a nearly clear background. If only one film is to be retained in a permanent file, a positive copy appears to be the best choice if it will serve the intended purpose.

### Inspection

Assurance that records are being preserved is obtained by a regular program of inspection. Details of sampling and inspection procedures are outlined in NBS Handbook 96.<sup>4</sup>

<sup>1</sup> For further details, see Current research on preservation of archival records on silver-gelatin type microfilm in roll form, by C. S. McCamy and C. I. Pope, J. Res. NBS 69A (Phys. & Chem.), No. 5, 385 (Sept.-Oct. 1965).

<sup>2</sup> The National Microfilm Association secured financial support for this research at NBS from the Bell & Howell Co., E. I. duPont de Nemours & Co., Dynacolor Corp., International Business Machines, Minnesota Mining & Manufacturing Co., Recordak Corp., University Microfilms, and the Xerox Corp.

<sup>3</sup> Formation of silver sulfide in the photographic image during fixation, by C. I. Pope, J. Res. NBS 64C (Engr. & Instr.), No. 1, 65 (Jan.-Mar. 1960).

<sup>4</sup> Inspection of processed photographic record films for aging blemishes, by C. S. McCamy, NBS Handbook 96 (1964). Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for 25 cents.

# EFFECT OF HIGH PRESSURE ON REFRACTIVE INDEX

## PHOTOELASTICITY THEORY VERIFIED

Direct measurements of the density coefficients of refractive index of several glasses and other crystalline materials have been made successfully at the NBS Institute for Materials Research. R. M. Waxler and C. E. Weir developed apparatus to permit these direct measurements at pressures up to 1,000 bars ( $10^8$  N/m<sup>2</sup>) in a study<sup>1</sup> of the effects of high pressure on refractive index. The data obtained verify F. Pockels' geometric theory of photoelasticity<sup>2</sup> and H. Mueller's physical theory.<sup>3,4</sup>

The results of this work indicate that uniform compression of crystalline solids has two effects on refractive index. First, by increasing density, it increases the number of scattering centers per unit volume and thus increases the index. Secondly, it increases interatomic forces, thus decreasing the size of the electron clouds (atomic polarizability) and refractive index. The net change in index is therefore governed by the predominating effect.

For potassium bromide, sodium chloride, and the glasses, all of which have weak interatomic forces, the index changes caused by density predominated. For diamond and magnesium oxide, which have strong interatomic forces, the polarizability decrease outweighed the density increase. In one substance, lithium fluoride, the two effects canceled each other.

### Experimental Procedure

In this study, specimens were prepared from 15 crystalline solids and 10 glasses. These were ground and polished into plates about 0.5 cm thick with plane, polished, nearly parallel faces. A layer of aluminum was vacuum deposited on the surfaces to increase their reflecting powers

and thus improve the visibility of interference fringes. The specimens were then immersed in a liquid-filled pressure vessel equipped with glass windows, and hydrostatic pressure was generated by compressing the liquid. Localized interference fringes between the two surfaces of the plate were viewed in reflection using collimated helium light ( $5875.62 \text{ \AA}$ ) at normal incidence.

As the hydrostatic pressure decreased plate thickness, the refractive index changed; the combined effects caused the interference fringes to shift. Measurements of the fringe count (number of fringes shifted), original index, and linear compression permitted calculation of the change in index. The sensitivity of these measurements to change in index was  $1 \times 10^{-5}$  and the refractive index values were reproducible within  $\pm 2 \times 10^{-5}$ .

For uniaxial crystals, each specimen was cut so that the surface plane contained the optic axis. A large polarizer which could be rotated was inserted between the light source and the window of the pressure vessel. Rotation of the polarizer produced two distinct fringe systems when the electric vector of the plane-polarized light was either perpendicular or parallel to the optic axis. The fringe shift for each separate system was measured and changes in index for both the ordinary and extraordinary rays were calculated.

### Experimental Results

The changes in refractive index varied from 0.02 for arsenic sulfide glass to zero for lithium fluoride under pressures up to 1,000 bars ( $10^8$  N/m<sup>2</sup>). The refractive indices of most materials increased with pressure, but those of diamond, magnesium oxide, zinc sulfide, stron-



*Collimated helium light ( $5875.62 \text{ \AA}$ ) at normal incidence was used to view localized interference fringes in diamond under hydrostatic pressure up to 1,000 bars ( $10^8$  N/m<sup>2</sup>).*

tion a titanate, and of both the ordinary and extraordinary rays of aluminum oxide decreased. With rutile, the ordinary ray index increased, while the extraordinary ray index decreased. The index of lithium fluoride did not change.

The measured changes in refractive index were compared to those predicted from the results of photoelastic studies. These changes agreed in the direction of change, and generally agreed in the amount of change. The decrease in refractive index for  $\text{MgO}$ ,  $\text{SrTiO}_3$ , and diamond predicted by Pockels was corroborated in this work.

The measured change in the index of diamond was compared to published data on the effect of hydrostatic pressure on the dielectric constant of diamond, and was found to be in close agreement. Use was made of the fact that with diamond the dielectric constant equals the square of the refractive index.

The relationship between the index of refraction and density is of interest because atomic interactions affect atomic polarizabilities. On each solid studied, an increase in hydrostatic pressure produced a net decrease in atomic polarizability. A constant trend in the data showed that unit change in polarizability per unit change in density was greater for substances with strong interatomic forces than for those with weak interatomic forces. In substances with strong interatomic forces, the change in index

is therefore governed by the change in polarizability and thus index decreases as hydrostatic pressure increases. This finding verifies Mueller's physical theory of photoelasticity.

### Temperature Versus Pressure

During this study the effect of temperature on the refractive indices of the same materials was also investigated. The change in index with change in temperature may be attributed to three factors: (1) A change in the number of scattering centers per unit volume, (2) a change in polarizability resulting from density change, and (3) a change in polarizability caused by temperature change exclusive of density change.

Generally, refractive indices that decreased with pressure, increased with temperature, and vice versa. For glasses, however, the refractive indices increased with both pressure and temperature. This result is attributed to a large temperature coefficient of polarizability coupled with a low coefficient of thermal expansion.

<sup>1</sup> For further information, see *Effect of hydrostatic pressure on the refractive indices of some solids*, by R. M. Waxler and C. E. Weir, *J. Res. NBS* 69A (Phys. & Chem.), No. 4, 325-333 (July-Aug. 1965).

<sup>2</sup> F. Pockels, *Weid. Ann. Phys.* 37, 151 (1889).

<sup>3</sup> H. Mueller, *Phys. Rev.* 47, 947 (1935).

<sup>4</sup> H. Mueller, *Physics* 47, No. 6, 179 (1935).



## STANDARDS AND CALIBRATION

### U.S. STANDARD FREQUENCY AND TIME BROADCASTS

WWV—2.5, 5.0, 10.0, 15.0, 20.0, and 25.0 MHz

WWVH—2.5, 5.0, 10.0, and 15.0 MHz

WWVB—60 kHz

Radio stations WWV (Greenbelt, Md.) and WWVH (Maui, Hawaii) broadcast signals that are kept in close agreement with the UT2 scale by making step adjustments of 100 ms as necessary. Each pulse indicates that the earth has rotated approximately 15 arcseconds about its axis since the previous one. Adjustments are made at

0000 UT (7:00 p.m., e.s.t.) on the first day of a month. There will be no adjustment on 1 October 1966. The pulses occur at intervals that are longer than 1 second by 300 parts in  $10^{10}$  due to an offset in carrier frequency coordinated by the Bureau International de l'Heure, Paris, France.

Radio station WWVB (Fort Collins, Colo.) broadcasts seconds pulses derived from the U.S. Time Standard (USTS) with no offset. Step adjustments of 200 ms are made at 0000 UT on the first day of a month when necessary. NBS directs that such adjustments be made in the scale at intervals to maintain the seconds pulses within about 100 ms of UT2. There will be no adjustment made on 1 October 1966.

*Members of the National Inventors Council meet with Vice President Hubert H. Humphrey in January 1966. Consisting of 23 prominent scientists and engineers, this Department of Commerce advisory panel is concerned with the processes of inventions, the work of inventors, and ways to provide more effective assistance to them through State, regional, and Federal invention programs. Left to right: Jacob Rabinow, Rabinow Electronics; Professor John C. Stedman, University of Wisconsin; J. Presper Eckert, Sperry Rand; Richard R. Walton, inventor; Jay W. Forrester, M.I.T.; Watson Davis, Science Service; Professor John Bardeen, University of Illinois; Costas E. Anagnostopoulos, Monsanto; Donald Newhouse, Portland OREGONIAN; William B. McLean, U.S. Naval Ordnance Test Station-China Lake; Narinder S. Kapany, Optics Technology; Samuel Ruben, Ruben Laboratories; Vice President Humphrey; Daniel V. DeSimone, Chief, NBS Office of Invention and Innovation and Executive Director of the Council; Willis H. Gille, Marquette Manufacturing; Dr. C. Stark Draper, M.I.T.; Martin Goland, Southwest Research Inst.; Chester Carlson, inventor; William Bollay, Stanford University; and, Leonard S. Hardland, NBS. Members not shown in the photograph are: Lawrence Biebel, attorney; Marvin Camras, Research Institute of I.I.T.; Frank E. Foote, Mine Safety Appliances Co.; John Hrones, Case Institute of Technology; Selman A. Waksman, Rutgers University; and, Brooks Walker, Shasta Forrest Co.*



# State Invention Shows

■ At a 1960 North Dakota inventors' exposition, a floating-beam plow that rides over rocks was exhibited by its inventor, a North Dakota farmer. Here it attracted the attention of an established manufacturer who later added it to his line. The plow now grosses a half million dollars a year for the company, and a new plant has recently been built to house the 50 employees engaged in its production. This is but one example of the way in which State invention expositions are today promoting economic development by bringing new products to the market place.

Last year more than 15 State invention exhibitions were held under the sponsorship of State agencies and local chambers of commerce. These shows gave inventors from all parts of the country an opportunity to demonstrate their inventions to potential manufacturers and distributors. The inventions exhibited covered a wide range—from a safety seat for babies to a collapsible camping trailer, from an exerciser for birds to a magnetic flux sensor for research use. An even greater number of such expositions are scheduled for 1966.

Most of these expositions have come about as the result of a State assistance program started in 1964 by the NBS Institute for Applied Technology, through its Office of Invention and Innovation (OI&I). An OI&I group under Leonard S. Hardland is now working actively with State

governments and local agencies to help them set up and run successful invention expositions. Besides providing advice and guidance to the organizing body, this group makes professional advice directly available to aid the inventors who attend in marketing their devices. Such assistance is particularly needed in States with less industry.

Before the OI&I program was begun, annual inventors' congresses had been held in only three States—in North Dakota beginning in 1957, and later in South Dakota and Minnesota. But the results obtained in these States had clearly shown the value of such meetings both in helping inventors to profit from their discoveries and in stimulating regional industrial activity. Recognizing their potential for advancing the Nation's technology, OI&I began a systematic effort to encourage these shows throughout the country.

The invention exposition program is directly in line with the basic mission of OI&I; namely, to help in the development of a national environment that is more conducive to technological innovation. Other activities carried on by OI&I as part of this mission include research on technological change, operation of an invention referral and information center, a design case program for engineering students, and provision of staff support for two Department of Commerce advisory panels—the National





# Shows Aid Industry

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Inventors Council and the Invention and Innovation Panel. The objective of the OI&I research program is to learn more about the processes of invention, innovation, and diffusion of technology, with particular emphasis on incentives and barriers to these processes and the impact of climate-setting Federal policies on them. The information center serves as a clearinghouse through which inventions submitted by private citizens for Government use are referred to other agencies of the Government; it also acts as a reference center for inquiries received from the public regarding inventions and inventors' problems. The engineering design case program is a new activity whose aim is to prepare and make available imaginative design cases, based on current projects of the Institute for Applied Technology, for use in developing the creative potential of students in engineering schools.

An inventors' exposition involves a 2- or 3-day meeting during which inventors may negotiate the sale or license of patents and arrange for the production and distribution of new inventions and new products. It also provides educational sessions—seminars and discussion groups—which assist the inventors in visualizing and coping with the many problems they face in promoting their inventions.

Upon request from a State government, OI&I will supply guidelines for the sponsoring agencies and examples of typical registration forms, programs, award certificates,

terminal questionnaires, and the like. OI&I will also seek and coordinate the assistance of other Government agencies such as the Patent Office and the Small Business Administration, and will assist in obtaining outstanding speakers from these agencies.

Invention expositions are usually held at fair grounds or in civic auditoriums. Here, all manner of inventions are displayed and explained in booths operated by the exhibitors. Normally, cash prizes and ribbons are awarded for several different classes of inventions. Although most of the inventors come from the host State, many exhibitors are from out of State; some may have attended shows in several States. At the North Carolina Inventors' Congress, held in Raleigh in May 1965, inventors were present from 26 different States as well as from Puerto Rico and Canada.

Besides meeting potential manufacturers and distributors, an inventor may benefit from the publicity given to his invention in the local press during an exposition. He also has an opportunity to assess the reaction to his invention as shown by representatives of the public who visit his booth. In this way he can judge the sales appeal of his device.

Figures compiled from the North Carolina exposition showed an attendance of 86,000 in the 3 days it was held. Of the 135 exhibitors, 21 stated that their inventions had

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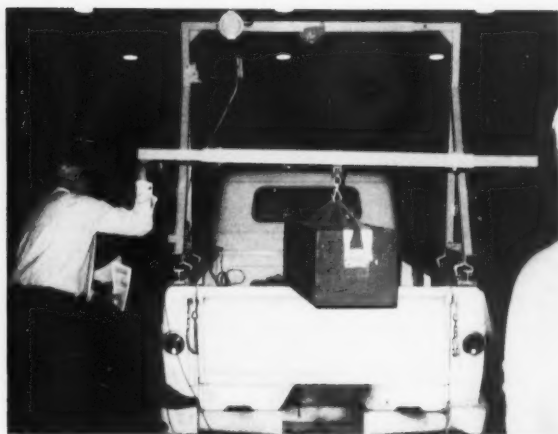
## STATE SHOWS *continued*

attracted enough attention to make the show worth while.

Sizable businesses may develop quickly from contacts made in this way. A push-button camping trailer, which was exhibited at the 1958 Minnesota invention exposition, is now being manufactured in a plant employing 65 persons; 900 units were produced in 1965. This trailer is kept low in height while under way on the road, but rises to full height at the touch of a button.

A scooter that travels woodland trails was exhibited at the 1962 Washington State inventors' exposition in Seattle. The inventor is now selling about 1,500 scooters a year for \$250 apiece.

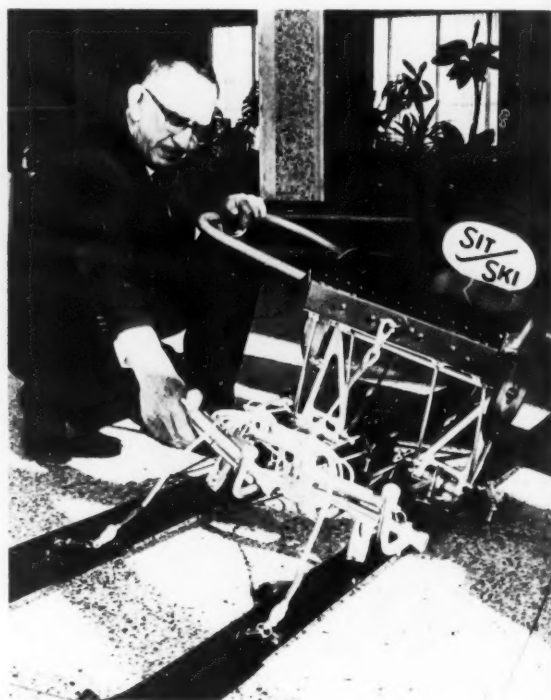
Other inventions that have profited from exhibition at State invention shows include a device for distinguishing slugs from coins in vending machines, an electric troller to be controlled by a fisherman on shore, a freezess water supply system for mobile homes and stock fountains, a gravity-return golf range, a remote-control chimney cap, a variable shotgun choke, an even-air ventilator, a hydraulic-squeeze bail loader, a patented fertilizer gun, and a "silent fisherman" for unattended fishing.



*A new design of truck loader is demonstrated at the Montana Inventors Congress held at Lewistown, Mont., in May 1966.*



*A new parking meter that stops meter looting is displayed at the Wisconsin Inventors and New Products Exhibition held at Green Lake, Wis., in May 1966.*



*Thomas R. Sherrod demonstrates his invention—a device designed for the skier who likes to sit down—at the Utah Inventors' Exposition held in March 1966 at Salt Lake City.*

# CONSTRUCTION OF NEW WWV BUILDINGS BEGUN

■ Construction is now under way on the NBS standards broadcast station WWV at Fort Collins, Colo. A contract was awarded early in May to a local builder, W. K. Livingston, for the construction of the transmitter building and administration building at the Fort Collins site. This is the last major contract affecting the move of WWV from Greenbelt, Md., to Fort Collins. According to present plans WWV will start broadcasting from its new site about December 1, 1966.

Because of the central location of Fort Collins, WWV signals from there will be received better throughout most of the continental United States than those from Greenbelt. No additional services are planned from the new location at this time.

The new transmitter building, located near the present WWVB-WWVL building, will be a T-shaped, cinder-block structure with a flat, steel girder roof. It will contain 6,880 square feet of work space, including a 4,200-square-foot transmitter and laboratory area, a 744-square-foot area for standby equipment, and a 961-square-foot machine shop and garage.

The 40×25-foot administration building will adjoin the present WWVB-WWVL transmitter building and will be a prefabricated metal structure conforming to it in appearance. This building will contain a lobby, the administrative offices for all three stations, a conference room, and facilities for displays and public briefings.

Four of the eight transmitters at the new location will be capable of radiating 10 kW of power and the other four, 2½ kW. Three of the 10-kW transmitters will operate continuously—at 5, 10, and 15 MHz—and one will be maintained as a standby for use at any of these frequencies. Three of the 2.5-kW transmitters will be operated at 2.5, 20, and 25 MHz and the fourth kept as a standby for these frequencies. The transmitter power amplifiers are of a commercial design and are being supplied by the Technical Materiel Corp. The transmitter drivers operating the power amplifiers are being made to NBS specifications.

Six of the eight antennas are half-wave, center-fed, vertically polarized, modified sleeve antennas from the Rohn Manufacturing Co., each operating at one of the six frequencies. The two standby transmitters will be connected to two identical monopole antennas, supplied by the Collins Radio Co., which can be used at any of the WWV frequencies.

Transmissions from WWV are now synchronized by means of phase-lock receivers tuned to the WWVL and WWVB transmissions from Fort Collins. Phase-lock receivers will not be needed when WWV is relocated: coaxial cables will connect it with WWVL and WWVB for continuous frequency and phase comparison.

The correct time has been established at WWV from portable electronic clocks that were set at the NBS Radio Standards Laboratory at Boulder, Colo., and carried, still running, to the Greenbelt transmitter. A similar procedure after the move will establish the correct time at all three stations simultaneously.

Most of the equipment presently in use at WWV is more than 15 years old and will be replaced with new and up-to-date equipment at Fort Collins. The new transmitters, programers, time code generators, and frequency control equipment will offer better precision than those currently in use.

Propagation time delay from Fort Collins will depend on where the receiver is located and will not differ from the time delay from Greenbelt by more than 9 msec. When the time comes for the changeover, the broadcasts on all frequencies from Greenbelt will be cut off at the instant that the transmitters at Fort Collins go on the air. Transmissions will continue on all frequencies without interruption so that no users will lose the signal.

Technical information about the services of NBS standards stations WWV, WWVH (Maui, Hawaii), WWVB, and WWVL can be obtained from NBS Miscellaneous Publication 236, Standard Frequency and Time Services of the National Bureau of Standards. The 1966 edition is available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, at a cost of 15 cents per copy. Each month the Standards and Calibration column in the TNB includes an item entitled U.S. Standard Frequency and Time Broadcasts that reports any changes in these services. (See p. 157 for the October 1966 announcement.)

*Left: this is one of two standby antennas recently installed at the new facilities for NBS radio station WWV. The coaxial transmission line (bottom) will connect with a transmitter in the new building, under construction to the left. Right: the standby antenna, fed by a 3½-inch coaxial transmission line, is examined by Hugh Stewart of the NBS staff. Transmitters in the new WWV building, under construction (background), will feed seven other antennas, one for each of the six transmission frequencies and another standby antenna. These antennas have been designed for ruggedness, compactness, and high efficiency.*

# ACCURATE PEAK PULSE VOLTAGE INSTRUMENTS

## for Low and Medium Ranges

■ In response to requests from industry and other government agencies, scientists at the Radio Standards Laboratory of the NBS Institute for Basic Standards have improved the accuracy of measurements of peak pulse voltages in the 5- to 100-V range. NBS engineers Paul A. Hudson and A. R. Ondrejka, of the Boulder (Colo.) Laboratories, developed an instrument system that has measurement uncertainties within 0.3 percent. This is more than an order of magnitude better than typical uncertainties of peak pulse voltmeters formerly available. The system includes a slideback voltmeter and a calibrated peak limiter; they can be used together for frequent checks of system accuracy.

### Progress in Measuring Pulses

Accurate measurements of low- and medium-level peak pulse voltages have recently become necessary in the development of high-speed computers and pulse-modulated communications systems. The appearance of more peak-reading voltmeters has resulted in a need for a new certification service at the Bureau in the range from 5 to 100 V. The system developed at the Bureau for this service consists of two instruments, one a refined slideback voltmeter and the other a limiter which standardizes pulses from a generator by means of cascaded Zener diodes.

The two instruments can be operated independently and can be readily intercompared, one measuring the pulse voltage output of the other without use of a transfer instrument. In initial tests the slideback voltmeter agreed with the Bureau's a-c and rf voltage standards within 0.1 percent at 100 Hz, within 0.3 percent at 10 and 30 MHz, and within 2.5 percent at 100 MHz. Agreement between the slideback voltmeter and pulse limiter was within 0.3 percent at several voltage levels. These instruments are believed to offer accuracies within 0.25 percent, which is considered to be adequate for calibrating the commercial instruments now available.

Further work planned includes extending the system ranges above 100 V and below 5 V, as well as improving accuracy for pulsewidths of as little as a few nanoseconds. Standards for peak pulse voltages as high as 100 kV have already been developed at the Institute's high voltage laboratories at Washington, D.C.

### The Slideback Voltmeter

The slideback voltmeter consists essentially of circuitry

for matching the peak being measured with an adjustable d-c voltage. The match is obtained by varying the voltage so that the peak being measured just merges with the base line on an oscilloscopic presentation. The d-c voltage is then read by means of a digital voltmeter which is accurate to within 0.05 percent. The overall system uncertainty is estimated to be 0.05 percent  $\pm 20$  mV.

The diode used in the slideback voltmeter is a critical component; the 1N270 semiconductor diode chosen consists of n-type germanium to which a gold wire has been welded to form a rectifying contact. The circuit used has an approximate rise time of only 10 nanoseconds. The circuit contributes only a slight error, due to an offset voltage which can be estimated from knowledge of the diode V-I curve and the detector sensitivity. A correction voltage (of the order of 40 mV) is added to the d-c voltage measurement, but it may vary as much as  $\pm 10$  mV due to thermal effects. This contributes to system uncertainty, but is independent of the input voltage level.

### Pulse Limiter

Accurate pulse voltages are obtained from a pulse generator by a circuit using Zener diodes to limit and regulate generator output pulses. The Zener diode is a special type which conducts no current until the applied voltage exceeds a certain value. Because of this characteristic it is widely used as a voltage reference in simple shunt circuits for precisely regulating alternating or direct voltages. The circuits now in use at the Bureau were designed to produce 5-100-V pulses which were found to be accurate within  $\pm 0.25$  percent in comparisons with d-c standards.

The diodes selected for this application have 10- and 50-W ratings and voltage-temperature coefficients of less than 0.1 percent/ $^{\circ}$ C. To minimize variation of output voltage with temperature, the designers of the circuit sought to limit diode power dissipation to only 1 W in order to confine variations in output voltage to 0.1 percent. They did this by careful selection of resistance values, use of two shunt circuits in series, and operating the circuits in liquid nitrogen for effective temperature stabilization. These measures have made the pulse limiter insensitive to variation in duty cycle from  $10^{-5}$  to 0.5, within the limits of the stated precision.

<sup>1</sup> Measurement standards for low and medium peak pulse voltages, by A. R. Ondrejka and P. A. Hudson, J. Res. NBS 70C (Engr. & Instr.), No. 1, 13-18 (Jan.-Mar. 1966).

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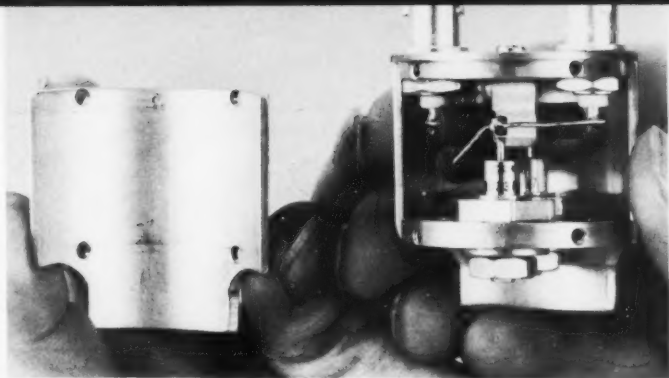
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*Left: Paul A. Hudson immerses pulse-clipping circuitry in a flask of liquid nitrogen to stabilize circuit performance. The console contains equipment for measuring peak pulse voltage and for generating pulses of known voltage, using the clipping circuit. Together, the instruments offer peak pulse voltage calibrations of improved accuracy. Right: peak voltage calibrations are made using pulses clipped to an accurately known level with this device. Its two Zener diodes and resistors are mounted in a "gondola" which is immersed in liquid nitrogen during operation, assuring that the clipping level will not be affected by thermal changes.*

## NMR CALIBRATION OF PERMANENT MAGNET STANDARDS

■ Measurements of magnetic fields are necessary for control and calibration of permanent magnet motors and generators, radar equipment, traveling wave tubes, magnetometers, and many other devices. To insure accuracy in such measurements, the measuring instruments used must often be calibrated at the location of the equipment. In many cases the calibration standard is a permanent magnet which in turn must be accurately calibrated, usually in a standards laboratory.

To meet the growing demand for the calibration of permanent-magnet standards, I. L. Cooter and R. E. Mundy, of the NBS Institute for Basic Standards, have recently adapted a nuclear magnetic resonance (NMR) technique to permanent-magnet standardization. This technique allows more accurate calibration of permanent magnets having fields in the range of 0.001 to 2 teslas.

The calibration procedure is based on the accurate measurement of the flux density in the gap of an electromagnet by an NMR magnetometer. This accurately known flux density is then compared with the flux density in the gap of the permanent magnet standard through the use of a suitable transfer instrument.

To measure flux density with the NMR magnetometer, a probe containing a sample of water is immersed in the steady, homogeneous magnetic field to be measured. By means of a surrounding coil, the water sample in the probe is also subjected to an alternating magnetic field at right angles to the steady field. Then the frequency of the alternating field is adjusted to equal the resonance frequency of the nuclei in the sample. At this frequency, which is directly proportional to the intensity of the steady field, there is an absorption of energy from the

exciting current. This energy absorption is observed by means of a cathode-ray oscilloscope and the resonance frequency is read from a digital counter. The flux density, in teslas, is then calculated from

$$B = \frac{\gamma}{4.25759 \times 10^7}$$

where  $\gamma$  is the resonance frequency in hertz, and  $4.25759 \times 10^7$  hertz/tesla is the gyromagnetic ratio of the proton divided by  $2\pi$ .

In the NBS nuclear magnetic resonance calibration, a measuring system consisting of a probe head and a measuring instrument is used as a transfer device between the reference field of a 7- or 12-inch electromagnet and the field of the permanent magnet standard.

The field supplied by the electromagnet is constant and homogeneous over a volume larger than the measurement probe head of the NMR magnetometer. The NMR probe head is placed near the center of this highly uniform magnetic field while the probe head of the transfer device is inserted in the gap of the permanent magnet and a reading is obtained. A sensitive digital voltmeter is used as a readout device for the transfer probe.

The transfer probe is then inserted in the gap of the electromagnet adjacent to the NMR probe. Next, the current from the power supply of the electromagnet is adjusted until the same reading is obtained on the digital voltmeter as when the transfer probe was in the gap of the permanent magnet standard. The frequency of the radiofrequency field applied in the NMR magnetometer is then varied until a resonance curve appears on the cathode-ray oscilloscope. This resonance frequency is measured by means of an electronic counter. Once the resonance frequency is obtained the flux-density existing in the gap of the electromagnet may be calculated by using the equation given above. This flux density is, of course, equal to that in the permanent-magnet gap.



# PRECISE TECHNIQUE MEASURES ABSOLUTE REFLECTANCE

■ Reflectance measurements are used extensively by research laboratories, producers of finished products, and by scientists in the space industries to examine and evaluate materials. To increase the accuracy of these measurements, the NBS Institute for Basic Standards has recently devised an improved method<sup>1</sup> for precision calibration of standards of hemispherical reflectance on an absolute basis instead of relative to magnesium oxide. The calibration procedure, developed by D. G. Goebel, B. P. Caldwell, and H. K. Hammond III, employs an auxiliary sphere coated with pressed powder for use with a double-beam, integrating-sphere type of spectrophotometer. The procedure is a modification of the one used by J. A. Van den Akker and associates at the Institute of Paper Chemistry.<sup>2</sup>

The inner surface of the auxiliary sphere is coated with any highly reflecting, highly diffusing material, usually barium sulfate or magnesium oxide. The reflectance of the coating used on the interior of the sphere is evaluated by measuring the reflectance of a flat plate provided with a coating identical to that of the sphere coating. The areas of the interior of the sphere and of the entrance port are computed from measurements of their diameters. The absolute reflectance of the flat plate is obtained by solving an equation involving instrumental measurements of the plate, the effective reflectance of the port in the sphere, and the ratio of the area of the port to the area of the sphere.

The measurements are made by using a two-beam integrating-sphere-type spectrophotometer. The effective reflectance of the sphere opening and the reflectance of the coated plate are each compared to a common sample; then the ratio of sphere-opening reflectance to plate reflectance is calculated. Once this ratio and the geometry of the sphere are evaluated, the absolute value of hemispherical reflectance of any specimen can be obtained.

In developing this procedure, the NBS scientists used

spheres of 10- and 15-cm diameters. The spheres were constructed of aluminum and each had a circular entrance port 2.54 cm in diameter.

Some coatings were applied by spraying the surface with a suspension of  $\text{BaSO}_4$  powder in alcohol. Other coatings were applied by pressing dry  $\text{MgO}$  or  $\text{BaSO}_4$  powder onto the surface. The pressed coatings provided more reproducible reflectance measurements. The pressed coatings were applied to the sphere wall to a depth greater than the necessary thickness, then scooped out with a jig to provide a coating about 2.5 mm thick. Pressed-on coatings provide three advantages over sprayed coatings: (1) higher reflectance, (2) greater uniformity, and (3) more accurate sphere dimensions.

Over a 3-year period, 22 separate sets of measurements were made utilizing these auxiliary spheres. Eight sets of data obtained with sprayed coatings were discarded because of nonuniformity in coating reflectance. The remaining 14 sets of data were used to determine the absolute value of a durable standard of hemispherical reflectance. Analysis of the results revealed that values of absolute reflectance could be reproduced with a standard deviation of 0.003.

The absolute  $6^\circ$ -hemispherical reflectance values for Vitrolite standards calibrated for use on the Hardy-type (General Electric) recording spectrophotometer are approximately 1 percent lower than the values relative to  $\text{MgO}$  previously given in NBS reports of calibration. The factors for converting relative-to- $\text{MgO}$  data to absolute data are the absolute  $6^\circ$ -hemispherical reflectance values of a 1-mm thick layer of  $\text{MgO}$ , and are tabulated for 10-nanometer (nm) intervals from 400 to 750 nm. To compute the absolute  $6^\circ$ -hemispherical reflectance of any NBS Vitrolite standard calibrated relative to  $\text{MgO}$ , one should simply multiply the reported Vitrolite reflectance for each wavelength by the tabulated value of absolute reflectance of a 1-mm thick specimen of  $\text{MgO}$  (table 1).





Jack D. Kuder measures the internal reflectance of an auxiliary sphere (lower right) by means of a double-beam integrating spectrophotometer.

The reflectance of the sphere is then used in a procedure to determine the absolute reflectance of a reference standard.

TABLE 1.\* Absolute 6°-hemispherical reflectance of smoked MgO 1 mm thick

Wavelength (nanometers)	Reflectance	Wavelength (nanometers)	Reflectance
400	0.989	600	0.991
	.990		.990
	.991		.990
	.992		.990
	.992		.990
	.992		.990
	.992		.990
	.992		.989
	.993		.989
500	.993	700	.988
	.993		.987
	.992		.987
	.992		.987
	.992		.987
	.992	750	.987
	.992		.987
	.992		
	.991		
	.991		

\*These data are based on 16 recent determinations of the absolute reflectance of an NBS Reference Vitrolite and 20 earlier determinations of its reflectance relative to 1-mm thick specimens of smoked MgO. Note that the data reported in Reference 1 are based on 12 determinations of absolute reflectance and therefore are slightly different.

<sup>1</sup> For further details, see Use of an auxiliary sphere with a spectrophotometer to obtain absolute reflectance, by D. G. Goebel, B. P. Caldwell, and H. K. Hammond III, J. Opt. Soc. Am. 56, 6, 783-788 (June 1966).

<sup>2</sup> Evaluation of absolute reflectance for standardization purposes, by J. A. Van den Akker, L. R. Dearth, and W. M. Shillcox, J. Opt. Soc. Am. 56, 2, 250-252 (February 1966).

## SYMPOSIUM ON CERAMIC MATERIALS

■ A 1-day symposium on ceramic materials and their properties will be held on Friday, October 14, 1966 at the National Bureau of Standards, Gaithersburg, Md. The symposium is jointly sponsored by the American Ceramics Society, the National Bureau of Standards, and the American Society for Testing and Materials.

The program is intended primarily for engineers and scientists whose specialties are in other fields. It will provide a concise introduction to ceramic materials and their properties, somewhat in the nature of a 1-day short course. Because it will provide a broad, contemporary perspective on the subject, the symposium will also be of interest to ceramists.

The committee in charge of arrangements consists of: P. M. Corbett, Glidden Co.; J. L. Pentecost, Melpar, Inc.; D. W. Robertson and W. S. Treffner, General Refractories Co.; and J. B. Wachtman, Jr., National Bureau of Standards.

Further information may be obtained by calling or writing Dr. Wachtman, 202-262-4040, extension 7703, National Bureau of Standards, Washington, D.C. 20234.

Payment of a \$5.00 registration fee by mail to Dr. Wachtman is requested.

The following program is scheduled:

"Scope of Modern Ceramic Materials"....	Cyrus Klingsberg, National Academy of Sciences
"Crystal Chemistry of Ceramics".....	H. F. McMurdie, American Society for Testing and Materials
"Phase Equilibria in Ceramic Systems"....	J. F. Schairer, Geophysical Laboratory, Carnegie Institute of Washington
"Thermal Properties of Ceramics".....	L. P. Domingues, U.S. Bureau of Mines
"The Value of ASTM to the Ceramics Industry".....	F. J. Mardulir, Vice Pres., ASTM
"Mechanical Properties of Ceramics".....	J. B. Wachtman, Jr., National Bureau of Standards
"Electrical Properties of Ceramics".....	J. L. Pentecost, Melpar, Inc.

Preprints of these papers are to be distributed at the symposium.



# NEWS

## Evaluation of Infrared Reference Spectra

Despite the large number of infrared spectra now available through commercial and literature sources, a great need exists for additional reference spectra. To help meet this need, the NBS Office of Standard Reference Data, together with the American Society for Testing and Materials, is exploring the feasibility of publishing a number of spectra from government and private collections. As such spectra must be evaluated and selected before they are published, the Coblenz Society has been commissioned to define specifications for them. The specifications are to guide evaluators in screening spectra submitted under the NBS program. These specifications should also prove useful to other spectroscopists in setting standards for their laboratories.

Under the Society's specifications, Class I spectra are "Standard Spectra." These spectra are of a sufficiently high quality to be acceptable as physical constants of the substances, under precisely defined conditions of measurement, and further refinements in spectrophotometric technique are not expected to change them significantly. Few, if any, spectra of this quality have as yet been produced, but they may be obtainable for condensed phase systems within 2 to 5 years. The unanimous opinion of the Board of Management of the Coblenz Society is that the specifications and format of such Standard Spectra should be defined by international agreement, and the Commission on Molecular Structure and Spectroscopy of the International Union of Pure and Applied Chemistry is studying this problem.

Class II spectra are reference spectra obtained on the best available commercial infrared grating spectrophotometers, operated at maximum efficiency under conditions consistent with acceptable laboratory practice. The purity of the compounds must be rigidly specified as to the type and quantity of any spectroscopically detectable impurity present.

Class II spectra differ from "Standard Spectra," because all available spectrophotometers distort the spectra to some extent, especially when operated under conditions acceptable for routine use. The specifications for Class II spectra apply only to the absorption spectra of condensed phase systems. Class II specifications may be upgraded as instrumental techniques improve; however, frequent

changes are to be discouraged and no major change should be necessary for the next few years.

Class III spectra are absorption spectra of individual compounds, polymers, and resins of known composition, obtained with sufficient accuracy to be useful in the identification of unknown materials. Only spectra that are superior to previously published spectra of the same compounds should be published. Spectra that fail to meet Class III specifications should not be published but may be useful to evaluators as comparison spectra. Laboratories submitting spectra to this collection are encouraged to meet Class II specifications as Class III is to be regarded primarily as a repository for usable data obtained before high resolution grating spectrophotometers were generally available.

The Coblenz Society emphasizes that the evaluators cannot be expected to determine reputed chemical structures from the infrared spectra alone. Their prime responsibility is to rule on the technical quality of the spectra and to check for gross incompatibility with the assigned structure. In some cases it may be desirable to refer the spectrum, after spectroscopic evaluation, to a chemist with special knowledge of the compound type; a panel of chemical specialists should be assembled for this purpose. Users of Class III spectra must be informed that these spectra may have come from large collections that were not compiled with this kind of definitive documentation in mind, so that some incorrectly assigned spectra may pass through the screening process.

The Coblenz Society recommends that spectra in Classes II and III be called "Reference Spectra" and be so designated in published charts and associated literature. It strongly deprecates the use of the word "standard" other than in association with spectra of Class I.

Copies of the specifications may be obtained from the Coblenz Society or from the Office of Standard Reference Data.

## Compilation of Atomic Transitions of Interest for Gas Lasers

Dr. W. R. Bennett, Jr., of the Physics Department, Yale University, is compiling tables of atomic transitions that are of interest in gas laser development. Such information in a compact, consistent form will expedite much laser research and development work. Dr. Bennett has analyzed all possible transitions for 50 atomic species between

known atomic energy levels (based primarily on tables by C. E. Moore of NBS) on the basis of selection rules and parity considerations. He is using these values, in combination with reports of experimental studies, to assign transitions to those lines for which laser oscillation has been observed. The resulting tables will be published by NBS in the NSRDS series. This work is part of the joint National Bureau of Standards-Advanced Research Projects Agency laser program.

### Improved X-Ray Wave Length Standards

Dr. J. A. Bearden of Johns Hopkins University, a member of the National Academy of Sciences-National Research Council Committee on Fundamental Constants, is compiling tables of x-ray emission wavelengths for the NBS Office of Standard Reference Data. In a similar compilation, Dr. Bearden previously provided values accurate to about 10 parts per million, although the available accuracy theoretically should be in the range 1 to 2 ppm.

Dr. Bearden has determined that a major source of error lies in non-uniformities of crystal structure of the crystals used in x-ray diffraction experiments. First, the arbitrary "x-unit" wavelength units based on different crystal materials (e.g., calcite versus rock salt) are not compatible or interconvertible, as previously assumed. Secondly, even a single natural crystal (such as calcite, a generally accepted standard) shows inhomogeneities from one area to another, or for different orientations of the crystalline planes in the same area.

Careful duplication of experiments using the same spot on a single crystal fixed in the same orientation has given precision in the 1 to 2 ppm range. Similar precision is being obtained with very high-purity synthetically grown silicon crystals. Results of this work, combined with results of some annihilation experiments at NBS, are expected to lead to substantial improvement in accuracy of values for several fundamental constants. Dr. Bearden's work shows how critical evaluation of numerical data can lead not only to greater accuracy of results, but also to more profound understanding of scientific phenomena themselves.

### Joint NBS-AEC Data Compilation Projects

The Office of Standard Reference Data supports data compilation projects and data center activities under a variety of financial arrangements. One such arrangement is joint support, under an interagency agreement, of an individual data center which is closely related to the mission of a specialized Government agency. Through agreement with the Atomic Energy Commission, NBS has completed arrangements for the continuing support of two data centers in fiscal 1967.

The first of these, the Atomic and Molecular Processes Information Center at Oak Ridge National Laboratories, is in its third year of operation with cooperative support

from AEC and NBS. This center, under the technical direction of Dr. C. F. Barnett, has published and distributed comprehensive annotated bibliographies on atomic and molecular collision processes involving heavy particle-heavy particle interactions. During fiscal 1966 a critical review and data tabulation program was initiated; the first topic selected was "Ionization by Heavy Particles," and a review monograph on this subject is nearly completed.

The second jointly supported data center is the Radiation Chemistry Data Center at the Radiation Laboratory of the University of Notre Dame. Under the technical direction of Dr. Milton Burton, this center is in its second year of activity. The collection and analysis of the scientific literature of radiation chemistry, which began last year, will continue this year. Articles are being indexed and plans are being developed for the extraction and machine storage of selected data. Work also began on a compilation on specific reaction rates of processes in aqueous systems.

### Chemical Kinetics Information Center

A Chemical Kinetics Information Center has been established at NBS under the direction of Dr. David Garvin. Its basic purpose is to provide rapid access to research on rates of chemical reactions. The center aids the NSRDS in the preparation of critically evaluated rate data, by supplying pertinent bibliographic information. The center has an index to over 11,000 papers and a file of reprints, both of which are publicly available at NBS, Gaithersburg (Md.). It also answers queries from the scientific and technical community about chemical kinetics—again by providing references to the original articles. Queries should be addressed to Chemical Kinetics Information Center, National Bureau of Standards, Washington, D.C. 20234.

The initial input to the center consisted mainly of the bibliographic collections of NBS staff members, and included the *Tables of Chemical Kinetics, Homogeneous Reactions*, issued as NBS Circular 510 and NBS Monograph 34. These tables cover much of the kinetics literature up to 1960 with emphasis on reactions in solution. In addition, the coverage of this phase of kinetics has been bolstered by accession of a collection of over 3,000 papers assembled by C. H. Stauffer of St. Lawrence College under the auspices of NBS, National Academy of Sciences-National Research Council, and the U.S. Air Force. This material was intended to supplement NBS Monograph 34. Another related collection, of over 2,000 papers on solvent effects in chemical kinetics, has been developed from a bibliography assembled by the late G. C. Akerlof as part of the NSRDS critical review program.

The largest collection, recently added to the center, was

*Continued*

## NSRDS NEWS *continued*

compiled by Adolph Hochstim of the Institute for Defense Analyses for the Advanced Research Projects Agency. It comprises over 5,000 documents covering rates and cross sections for gas phase reactions between small molecules, ions, and free radicals.

In the future, the center hopes to serve as a depository for and as a rapid reference source of information on chemical kinetics. A long-term goal is to evaluate and compile rate data with particular emphasis on those areas in which a critical national need exists.

### Excerpts from Statement of J. Herbert Hollomon on NSRDS\*

... The present lack of a comprehensive, effective standard reference data system costs the Nation hundreds of millions of dollars each year. This is so because the work that an integrated, comprehensive, national standard reference data system could do is already being done. Nearly every member of the technical community does part of the job himself—piecemeal, uncoordinated, and usually less effectively than if done by an expert. Properly operated at full potential, a National Standard Reference Data System could return \$20 to \$200 for each dollar spent on it.

The products of the National Standard Reference Data System are valuable to the technical community because numerical data are made both more readily accessible and more reliable. Dollar benefits result from: (1) Savings of time of users in searching through scientific reports for numerical data; (2) savings of time in evaluating and selecting most reliable answers from among those found in scientific reports; (3) savings of time and materials spent in unnecessary measuring of properties of substances for which the data actually reported in the literature could not readily be found; (4) savings of time, equipment, and materials through use of better (more reliable) data.

Some of these savings—those in the first two categories—can be estimated quantitatively. An illustration is the example of NBS Circular 500, "Selected Values of Chemical Thermodynamic Properties," issued originally in 1952 and now being revised. This volume contained several thousand values of certain fundamental thermodynamic properties of all the elements, all inorganic compounds for which data were available, and organic compounds containing one or two carbon atoms. Over 7,000 copies of this book were sold—to scientists and engineers in academic laboratories, industrial laboratories, and government laboratories. Using conservative assumptions about the number of persons using each volume and the amount of time saved at each use because the individual did not have to search and evaluate for himself, we estimate that the equivalent value of this one

*Mrs. Ruth Brody of the Chemical Kinetics Data Center staff uses an automatic punch indexing apparatus to punch locator holes in indexing cards containing bibliographical information. Behind her, Francis Westley removes an indexing card from one of the files.*



volume to the economy of the United States has been \$50 million. The cost of producing this publication was about \$250,000. The ratio of benefits to cost in this case is 200 to 1—an unusually favorable ratio.

It is this type of estimate, which can be made for numerous similar works now in existence, which leads us to the conclusion that each dollar spent on producing standard reference data will save the economy \$20 to \$200. I must point out that these savings will be difficult to find. They will be made up of thousands of small timesavings, equivalent to a few dollars here and a few dollars there. No one will file a cost-saving report with his administration listing an item of \$30 for 3 hours that he didn't spend in the library because a compilation of evaluated data was available to him.

The estimates just described do not take into account the value to the economy of the availability of better data. These values are inestimable. How would one determine, for example, how many manufacturing plants had to be designed with broader tolerances because the available data were less reliable than they might have been? How does one determine how many missile shots have failed because incorrectly evaluated data were used in the design of some component? Although such incidents cannot be definitely identified, every scientist and engineer is confident that they occur and that they are costly indeed.

Important as such benefits are, they are perhaps matched in importance by the guidance provided to measurement practices by the data compilations. The thorough critical evaluation of sources of uncertainty in measurement technique inevitably leads to an upgrading of the quality of subsequent measurements in laboratories all over the country. This effect has already begun to be felt as a result of the NBS program. Further, by pointing out gaps in the availability of data and by identifying key properties for which higher precision is required, data evaluations serve as a means for an experimentalist to determine which measurements deserve high priority in his program . . .

\*Assistant Secretary of Commerce for Science and Technology.



# PUBLICATIONS of the National Bureau of Standards\*

## PERIODICALS

*Technical News Bulletin*, Vol 50, No. 8, August 1966. 15 cents. Annual subscription: \$1.50. 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

*Journal of Research of the National Bureau of Standards*

*Section A. Physics and Chemistry*. Issued six times a year. Annual subscription: Domestic, \$5; foreign, \$6. Single copy, \$1.00.

*Section B. Mathematics and Mathematical Physics*. Issued quarterly. Annual subscription: Domestic, \$2.25; foreign, \$2.75. Single copy, 75 cents.

*Section C. Engineering and Instrumentation*. Issued quarterly. Annual subscription: Domestic, \$2.75; foreign, \$3.50. Single copy, 75 cents.

## CURRENT ISSUES OF THE JOURNAL OF RESEARCH

*J. Res. NBS 70A* (Phys. and Chem.), No. 4 (July-August 1966), \$1.00.

Elastic constants of synthetic single crystal corundum. W. E. Tefft.

Phase equilibria as related to crystal structure in the system niobium pentoxide-tungsten trioxide. R. S. Roth and J. L. Waring.

Properties of aqueous mixtures of pure salts. Thermodynamics of the ternary system: water-calcium chloride-magnesium chloride at 25 °C. R. A. Robinson and V. E. Bower.

Properties of aqueous mixtures of pure salts. Thermodynamics of the ternary system: water-sodium chloride-calcium chloride at 25 °C. R. A. Robinson and V. E. Bower.

Use of the consistency check in the vector verification method. A. D. Mighell, and R. A. Jacobson.

Self-reversal in the spectral lines of uranium. D. D. Laun.

Oscillator strengths for ultraviolet lines of Fe I. C. H. Corliss and B. Warner.

*J. Res. NBS 70B* (Math. and Math. Phys.), No. 3 (July-September 1966), 75 cents.

Error bounds for asymptotic solutions of differential equations. I. The distinct eigenvalue case. F. Stenger.

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On the approximation of functions of several variables. B. Mond and O. Shisha.

Finding a rank-maximizing matrix block. A. J. Goldman and M. Newman.

On certain discrete inequalities and their continuous analogs. A. M. Pfeffer.

## OTHER NBS PUBLICATIONS

Periodicals and serials received in the Library of the National Bureau of Standards as of October 1965. N. J. Hopper, NBS Misc. Publ. 274 (July 1, 1966). 50 cents. Supersedes Mono. 57.

Cooperation, convertibility, and compatibility among information systems: A literature review. M. M. Henderson, J. S. Moats, M. E. Stevens, and S. M. Newman, NBS Misc. Publ. 276 (June 15, 1966), \$2.00.

Atomic transition probabilities. Hydrogen through neon, W. L. Wiese, M. W. Smith, and B. M. Glennon, NSRDS-NBS4, Vol. I (May 20, 1966), \$2.50.

Hosiery lengths and sizes excluding women's, CS46-65 (Dec. 31, 1965), 15 cents. Supersedes CS46-49.

Quarterly radio noise data March, April, May 1965, W. Q. Crichlow, R. T. Disney, and M. A. Jenkins, NBS Tech. Note 18-26 (May 2 1966), 45 cents. (Formerly the Central Radio Propagation

Laboratory of the National Bureau of Standards, now the Environmental Science Services Administration, Boulder, Colo.)

Transistorized building blocks for data instrumentation, P. G. Stein, NBS Tech. Note 268 (May 28, 1966), 60 cents.

Status report, National Standard Reference Data System April 1966, Ed. E. L. Brady, NBS Tech. Note 289 (June 1, 1966), 50 cents.

Calculation of the admittance of a parallel plate capacitor containing a toroid-shaped sample, E. G. Johnson, Jr., NBS Tech. Note 336 (Apr. 21, 1966), 30 cents.

Advances in ionospheric mapping by numerical methods, W. B. Jones, R. P. Graham, and M. Leftin, NBS Tech. Note 337 (May 12, 1966), 45 cents. (Formerly the Central Radio Propagation Laboratory of the National Bureau of Standards, now the Environmental Science Services Administration, Boulder, Colo.)

## PUBLICATIONS IN OTHER JOURNALS

*This column lists all publications by the NBS staff, as soon after issuance as practicable. For completeness, earlier references not previously reported may be included from time to time.*

### CHEMISTRY

A quick, direct method for the determination of activation energy from thermogravimetric data, J. H. Flynn and L. A. Wall, *Polymer Letters* 4, Pt. B, No. 5, 323-328 (May 1966).

A standard reference material for Mössbauer spectrometry of iron and its compounds, J. J. Spijkerman, F. C. Ruegg, and J. R. DeVoe, *Technical Reports Series 50, Applications of the Mössbauer Effect in Chemistry and Solid-State Physics*, pp. 254-259 (International Atomic Energy Agency, Vienna, Austria, 1966).

Action of zinc dust and sodium iodide in N,N-dimethylformamide on contiguous, secondary sulfonyloxy groups: a simple method for introducing nonterminal unsaturation, R. S. Tipson and A. Cohen, *Carbohydrate Res.* 1, No. 4, 338-340 (1965).

Phase rule considerations and the solubility of tooth enamel, W. E. Brown and B. M. Wallace, *Ann. N.Y. Acad. Sci.* 131, Art. 2, 690-693 (Sept. 30, 1965).

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\*Publications for which a price is indicated are available by purchase from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402 (foreign postage, one-fourth additional). The NBS nonperiodical series are also available from the Clearinghouse for Federal Scientific and Technical Information, Springfield, Va. 22151. Reprints from outside journals and the NBS Journal of Research may often be obtained directly from the authors.

## CLEARINGHOUSE BIBLIOGRAPHIC JOURNALS\*\*

*U.S. Government Research & Development Reports*. Issued 24 times a year. Annual subscription: Domestic, \$30; foreign, \$37.50. Single copy, \$2.25.

*Government-Wide Index to Federal Research & Development Reports*. Issued 24 times a year. Annual subscription: Domestic, \$22; foreign, \$27.50. Single copy, \$1.75.

*Technical Translations*. Issued 24 times a year. Annual subscription: Domestic, \$12; foreign, \$16. Single copy, 60 cents.

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